



THE UNITED STATES PATENT & TRADEMARK OFFICE

In re Application of Tresch et al.

Serial No. 09/936,432

Filed: September 13, 2001

For: AZOXY DYES AND COPPER COMPLEXES THEREOF

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DECLARATION

I, Thomas Gessner, Dr. rer. nat., a citizen of the Federal Republic of Germany and residing at Gundolfstrasse 1, 69120 Heidelberg, Germany, declare as follows:

I am a fully trained chemist, having studied chemistry at the Universities of Mainz and Munich, Germany, from 1975 to 1986;

I was awarded my doctor's degree by the said university in 1986:

From 1987 to 1988 I was a post-doctoral fellow at the University of Chicago;

I joined BASF Aktiengesellschaft of 67056 Ludwigshafen, Germany, in 1988, and have since been working in the field of organic dyestuffs and their preparation.

I am familiar with the invention disclosed and claimed in application serial No. 09/936,432;

I have read the Office Action of April 4, 2003 and the references cited therein by the Examiner.

I Experiments for reproducing prior art

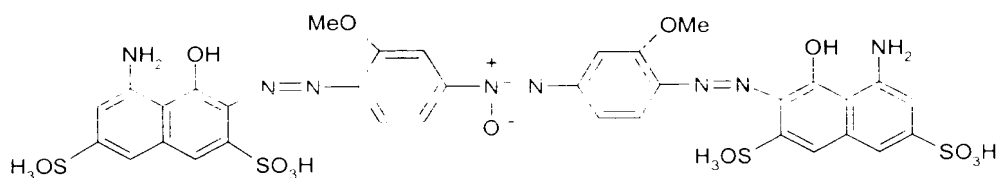
I have performed example 3 of US 2,203,196 and analyzed the resulting blue dye. I have chosen mass spectrometry (ESI, Electron Spray Ionization) as analytical method which is a very sensitive method for detecting even traces of formed dye products. The blue dye

had no mass peak of 920 which is expected for the formula given in US 2,203,196. Not even traces of a mass peak of 920 could be detected at.

I also performed synthesis example 3 of US 5,609,796 and repeated the example varying the reaction conditions with respect to the amount of sodium hydroxide in the coupling. For the strong alkaline conditions in the coupling step I chose different amounts of sodium hydroxide up to 94 parts of sodium hydroxide. The resulting methoxy substituted dye had a mass peak of 498 instead of 948. No traces of the expected peak of 948 could be detected.

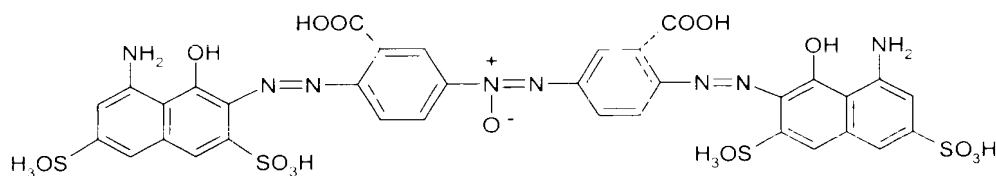
## II Investigation of dyeing properties

In order to compare the closest prior art compounds with compounds of Application serial No. 09/936,432, I selected the following dyes:



Dye 1

(Appln. Ser. No. 09/936,432)



Dye 2

(CH 172368, Ex. 1)

Both dyes were prepared in the same manner and were of equal purity when used for dyeing. Both dyes were dissolved in pure water. They were dyed to a 1/6 standard depth coloring. The coloristic data of the dyeing were evaluated by a color-measuring instrument. The measuring results were corrected by calculation to equal standard depth.

#### Dyeing procedure (woodfree unsized)

The liquid formulation of the dye was added to the dye-bath consisting of 5 g of bleached sulfate cellulose pulp (70% pine and 30% birch). It was stirred for 15 minutes at room temperature. The pulp was then diluted with water to a consistency of 1,5 g/l. From this pulp a sheet with a sheet weight 160g/cm<sup>2</sup> was made with a Rapid-Köthen-sheet forming device. After drying the coloristic data were measured. The higher the C\*-value the higher is the brilliance.

#### Dyeing procedure (woodfree sized)

The dyeing was performed analogously to the woodfree unsized dyeing procedure. Additionally 1 % paper making rosin and 3 % aluminium sulphate was added after the addition of the dye. The pulp was stirred for 15 minutes.

#### Substantivity

A measure for the substantivity of a dye is the grade of coloring of the waste water. A sample of the collected waste water from the sheet forming process was taken and the solid matter was separated by centrifugation. The resulting waste water was rated visually in a test tube.

Table 1: Dyeing of pulp woodfree unsized

	Hue H	Brilliance C*	Amount calc. pure dye	Waste water
Dye 1	244.21	27.11	0.19 %	very weak
Dye 2	261.87	24.99	0.66 %	weak (with red shade)

$$\Delta C (\text{Dye 2} - \text{Dye 1}) = -2.1$$

Table 2: Dyeing of pulp woodfree sized

	Hue H	Brilliance C*	Amount calc. pure dye	Waste water
Dye 1	248.79	26.07	0.18 %	very weak
Dye 2	261.67	16.58	0.78 %	weak (with red shade)

$$\Delta C (\text{Dye 2} - \text{Dye 1}) = -9.5$$

From the amount of dye used it is clearly seen that Dye 1 yields a three times stronger blue dyeing than Dye 2. In case of dyeing woodfree unsized pulp Dye 1 is 9.5 grades more brilliant than Dye 2. As can be seen from the exhibit Dye 1 yields a brilliant blue dyeing, the waste water being stained very weak.

In my opinion these results are truly surprising and could not have been foreseen by the skilled artist.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information or belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed at 67056 Ludwigshafen, Germany, this 28<sup>th</sup> day of August 2003.



Signature of Declarant